1053852 9

PATENT SPECIFICATION

(11) 1 458 222

1 458 222

(21) Application No. 51455/75 (22) Filed 12 March 1973
(62) Divided out of No. 1458221
(23) Complete Specification filed 4 March 1974
(44) Complete Specification published 8 Dec. 1976
(51) INT CL² H05B 5/16
(52) Index at acceptance
H5H 2G2B 2GX2 3S

(19)

(72) Inventors LYNDON JAMES MILES and IVOR WYNN JONES

(54) IMPROVEMENTS IN OR RELATING TO FURNACES FOR THE PRODUCTION OF TUBULAR OR CYLINDRICAL CERAMIC ARTICLES

(71) We, THE ELECTRICITY COUNCIL, a British Body Corporate, of 30 Millbank, London, SW1P 4RD, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to furnaces for the continuous production of tubular or disc-shaped ceramic articles by a sintering process in which compressed powder material is traversed through a furnace.

According to this invention a furnace for the production of tubular or disc-shaped ceramic articles comprises an open-ended tube through which the articles to be heated may be passed, with heating means for heating the tube, which heating means comprise an induction coil around a susceptor block, said open-ended tube passing through said block, and means for continuously rotating said tube about its axis.

The articles to be fired are moved through the tube at a uniform rate. A single article may be fired by pushing it through the furnace with a push rod. The rotation of the furnace tube ensures an even temperature. Also it causes the articles to roll around the inside surface of the furnace tube so helping to keep them straight and cylindrical.

This furnace has particular application to the sintering of β-alumina ceramic articles. β-alumina ceramic is a material having a nominal composition by weight of 5% Na_2O, 95 % Al_2O_3. The amount of sodium oxide in practice can range from 5 up to 10%. It may also contain oxides of magnesium and/or lithium. The material can be sintered in the temperature range 1550 to 1900°C. It is used in sodium sulphur cells and other electrochemical devices requiring passage of sodium ions. Desirable properties for this material in such applications include high density, imperviousness to helium gas and close control of composition and properties throughout the bulk and particularly right up to the surface of the material. The material may typically be required in the form of long thin wall tubes with or without a closed end or in the form of discs.

As is described in our earlier British Patent No. 1297373, articles of β-alumina ceramic may be produced by forming shapes of compressed powder of the required composition and moving these shapes through a tubular furnace so that a short length of the material is raised to the sintering temperature, the movement being continuous so that the heated zone is gradually moved along the length of the material to be fired. In that specification, there is described an induction furnace having a susceptor block with a fixed furnace tube extending through the susceptor block.

In use, the articles are moved at a uniform rate through the furnace so that a short length of material is raised to the sintering temperature with the heated zone gradually moving along the length of material to be fired. With this method, a very short heating and sintering time can be employed as described in the aforementioned Specification No. 1297373. By choice of the length of furnace, rate of traverse and firing temperature, close control of grain size can be obtained as described in that specification.

As is described in the specification of Application No. 11835/73 (Serial No. 1458221) out of which the present application is divided, as the unfired material is heated on entering the furnace it gives off water vapour and a small amount of sodium oxide vapour.

The furnace is preferably sloped upwardly with respect to the horizontal in the direction of movement of the articles or means are provided for forcing air to flow through the furnace tube in this direction.

The water vapour is thus carried forward through the furnace. The water vapour cannot recondense on the articles entering the firing zone and thereby accumulate in the furnace to reach critical concentration on the unfired material. By the evaporation of the small amount of sodium oxide from the incoming articles, a stable sodium oxide-rich atmosphere is produced and maintained in the firing zone. This is in contrast to the conditions which would occur, for example, if the gas flow was in the opposition direction to the movement of the article. In that case, the water vapour would recondense on the cold incoming material. The water concentration would tend to build up towards a critical value and cause the incoming tubes to break up in the furnace. The conditions in the sintering zone would be non-equilibrium and the properties of the ceramic material would deteriorate as the article or run of successive articles proceeds through the furnace.

For controlling the temperature in the furnace, conveniently there is provided a second tube through said susceptor block with open ends, a radiation pyrometer being arranged for observing a test element within said second tube. This test element may be of any suitable material (e.g. recrystallised alumina) such that temperature variations may be observed by the pyrometer. The temperature of the test element need not be necessarily the same as the ceramic article being sintered although preferably the furnace is arranged so that test element conditions are, as closely as possible, similar to those for the article being fired. However, variations of temperature of the test article will correspond to variations of temperature of the ceramic material being sintered and hence the output of the pyrometer may be used for controlling the temperature of the furnace. Preferably an automaic control system is provided for this control of temperature.

In one convenient arrangement, the furnace tube through which the ceramic articles are traversed and the tube containing the test article are symmetrically disposed on opposite sides of the axis of the induction heating coil. In such an arrangement preferably the continuously rotated tube containing the ceramic to be sintered, referred to hereinafter as the firing tube, is arranged within a second stationary tube of slightly greater diameter extending through the susceptor block and furnace. With such a construction it has been found possible to control the sintering zone temperature readily to an accuracy of ±5°C. Depending on the requirements, during operation the sintering zone may be maintained at any required temperature between say 1550 and 1900°C., typically at 1700°C.; each portion of the material to be fired may typically remain in the sintering zone for less than two minutes.

In the following description, reference will be made to the accompanying drawing which is a diagrammatic section through one embodiment of an induction furnace for sintering β-alumina ceramic.

Referring to the drawing, there is shown a furnace for the sintering of tubular β-alumina ceramic articles comprising an induction coil 10 energised from an alternating current generator 11 typically operating at 450 kHz. The coil 10 is a helical coil, typically only a few inches long and surrounding a graphite susceptor block 12. The coil 10 and susceptor block 12 are contained within an asbestos box forming a housing 13 which box is filled with bubbled alumina insulation, indicated diagrammatically at 14. Extending through the block 12 parallel to the axis of the coil 10 are two bores which are arranged symmetrically with respect to the axis of the coil. The first bore contains a refractory tube 15 which is open-ended and extends through the end walls of the asbestos box 13. The second bore through the susceptor block 12 is of larger diameter than the first bore and contains a fixed open-ended refractory tube 16 within which is a rotatable tube 17 of refractory material, e.g. alumina, for containing the article to be sintered. This tube 17 is carried in bearings 18 and continuously rotated by drive means indicated diagrammatically at 19 to equalise the temperature around the firing zone when the furnace is in operation. Typically rotation rates of 30 to 60 r.p.m. have been employed. The tube 15, when in use, contains a suitable refractory article indicated diagrammatically at 20, typically formed of recrystallised alumina, for observation by a total radiation pyrometer 21 for temperature control purposes. The output from the pyrometer 21 may be fed to a monitor 22 and/or to a power control unit 23 controlling the power output of the generator 11.

To preserve a stable soda-rich atmosphere required for firing β-alumina ceramic articles, the rotating tube 17 should be impervious to the sodium oxide vapour. If the material of the rotating tube reacts with sodium oxide vapour, then the rate of this reaction should be so low that sodium oxide depletion does not occur. It was found, for example, that sodium oxide did react slowly with the recrystallised alumina tube used in the experiments referred to below, converting it into β-alumina. The reaction rate was quite low, the firing tube

gaining 20 mg in weight for each 40 g tube which was fired. The loss from the articles being fired was less than 1% of the sodium oxide present. However the conversion of the firing tube to β-alumina is accompanied by a volume change so that this tube no longer remained impervious. For this reason only about 20 metres of tube could be fired before replacing the rotating tube 17; such replacement can be effected however without cooling the furnace.

Means (not shown) are provided for moving the articles to be fired continuously through the furnace at a uniform rate. A continuous air flow through the tube 17 of the furnace is obtained in operation by sloping the furnace upwardly in the direction of movement of the articles. The optimum angle of slope may be determined empirically; it is typically between 4° and 10° to the horizontal. Using convention in this way provides a very simple and reliable means of obtaining the required air flow; obviously however a forced air flow could be obtained in other ways without sloping the furnace.

Articles are traversed through the furnace in the upward sloping direction; a succession of articles may be moved through the tube, each being pushed by the next article in succession. Single articles may be moved through the furnace using a push-rod.

Reference may be made to the specification of co-pending Application No. 11835/73 (Serial No. 1458221) for a fuller description of examples of the manufacture of β-alumina ceramic tubes using the above-described furnace.

WHAT WE CLAIM IS:—

1. A furnace for the production of tubular or disc-shaped ceramic articles comprising an open-ended tube through which the articles to be heated may be passed, with heating means for heating the tube, which heating means comprise an induction coil around a susceptor block, said open-ended tube passing through said block, and means for continuously rotating said tube about its axis.

2. A furnace as claimed in claim 1 and having means for forcing air to flow through the tube.

3. A furnace as claimed in claim 1 and arranged with the tube sloping upwardly with respect to the horizontal.

4. A furnace as claimed in claim 3 wherein the tube slopes upwardly at an angle of 4 to 10°.

5. A furnace as claimed in any of the preceding claims wherein said susceptor block is a block of graphite.

6. A furnace as claimed in any of the preceding claims wherein, for controlling the temperature in the furnace, there is provided a second tube through said susceptor block with open ends, and wherein a radiation pyrometer is arranged for observing a test element within said second tube.

7. A furnace as claimed in claim 6 wherein automatic control means are provided responsive to said radiation pyrometer and arranged to control the power supply to said furnace.

8. A furnace as claimed in any of the preceding claims wherein the tube through which the ceramic articles are traversed and the tube containing the test element are symmetrically disposed on opposite sides of the axis of the induction heating coil.

9. A furnace as claimed in any of the preceding claims wherein the aforesaid open-ended tube which is continuously rotated, is arranged within a stationary tube of slightly greater diameter extending through the susceptor block and furnace.

10. A furnace for the production of sintered ceramic articles substantially as hereinbefore described with reference to the accompanying drawing.

BOULT WADE & TENNANT
Chartered Patent Agents
34 Cursitor Street,
London, EC4A 1PQ

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1976.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

1458222 COMPLETE SPECIFICATION

1 SHEET This drawing is a reproduction of the Original on a reduced scale